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Development of Scale to Predict Effect of Three Alkali Activated Solution on Compressive Strength and Microstructure of Geopolymer Based Fly Ash

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Abstract

This research aimed to develop a scale to predict the effect of alkali-activated solution on compressive strength and microstructure of geopolymer based fly ash. The starting material for geopolymer synthesis was fly ash obtained from Mea Moh electricity thermal power plant. Sodium hydroxide 10M, sodium silicate solution and calcium hydroxide solution were used as alkali-activated solutions for geopolymer. The proportions of alkali -activated in the geopolymer mixture were calculated by simplex lattice design. The compressive strength of geopolymer was investigated at the age of 7, 14 and 28 days. The surface area of the geopolymer samples was characterized by OM technic. The results of compressive strength were input into a Minitab program to construct the scale for predicting and plotting the mixture contour. The results showed that the compressive strength of geopolymer had a range between 0 and 200 ksc. Using sodium hydroxide and sodium silicate solution to be alkali activated solution for geopolymer provided the highest compressive strength. It is worth mentioning that sodium hydroxide is a strong alkali hydroxide which dissolved Si and Al from fly ash; additionally, geopolymer could also be formed from free silica in the sodium silicate solution. On the other hand, geopolymer synthesized with calcium hydroxide solution presented the lowest compressive strength due to the solution being a less strong alkaline to dissolve Si and Al from fly ash. The equation for predicting the compressive strength in ksc of geopolymer with three alkali-activated solutions is $Y = 202.49NH-3.63NS+43.20CH+461.24(NH*NS)-61.81(NH*CH)+559.15(NS*CH)+45.98(NH*NS*CH)$ where Y is compressive strength of geopolymer in ksc.

Keywords : Geopolymer; compressive strength; predict equation

Introduction

Geopolymer is a aluminosilicate materials such as fly ash and metakaolin which can be synthesized using fly ash and alkali hydroxide such as sodium hydroxide (NaOH). In addition, it can be added more silicate compounds to enhance compressive strength by adding sodium silicate solution (Na_2SiO_3). The reaction develops when alkali solution dissolves Si and Al to form free of tetrahedral of Si-OH and Al-OH. Then, water gradually split out, tetrahedral of Si-OH and Al-OH are alternately link by sharing oxygen atom. Generally, there are three types of geopolymer structure which are polysialate (Al-O-Si), polysialate-siloxo (Al-O-Si-O-Si-O) and polysialate-disiloxo (Al-O-Si-O-Si-O-Si-O). The mechanical properties of geopolymer is depended on Si:Al ratio. The Si:Al ratios as 2:1 was applied to low CO_2 cemen&concrete and radioactive&toxic waste encapsulation. It was found that calcium (Ca) contains some starting materials of geopolymer such as high calcium fly ash, slag. Calcium can react with silicon form calcium silicate hydrate which similar from cement hydration reaction and calcium geopolymer by geopolymer reaction and provide compressive strength [1].

The alkali activated solution is an important factor to develop geopolymer reaction. NaOH solution, KOH solution and NaOH solution with sodium silicate solution are usually used to be alkali activated solution for geopolymer [2-8]. High concentration of NaOH between 10-14 M provide good mechanical properties due to alkali hydroxide (OH-) help to leach Si and Al from starting materials to form geopolymer products [2-3]. Sodium silicate solution consist of SiO_2, Na_2O and H_2O. Soluble silica easily reacted with other compounds in geopolymer mixture while Na_2O is alkali solution in system. Sodium (Na) is a charge balance of aluminium (Al) to become tetrahedral products in geopolymer matrix [1, 7-8]. In addition, there are other types of alkali such as calcium hydroxide ($Ca(OH)_2$) which can be added in mixture to enhance compressive strength by pozzolanic reaction. Some researchers found that external calcium and internal calcium can add in geopolymer, the products of this sources are calcium silicate hydrate (CSH) and/or calcium aluminate silicate hydrate (CASH). It can help to enhance compressive strength of geopolymer [9-11].

Thus, this research is aims to study effect of alkali solution on mechanical property of geopolymer. 10 M NaOH, Na_2SiO_3 and $Ca(OH)_2$ were selected to be alkali solutions. The mixing proportion was designed by simplex lattice design and constructed the scale for predicting and plotting the mixture contour by Minitab program. The results presented the application of alkali solution to geopolymer materials to produce qualified construction product.

Methodology

Materials and solution

Fly ash was obtained from Mea Moh electricity thermal power plant, Lampang province. 10 molar NaOH, sodium silicate solution (Na_2SiO_3) and $Ca(OH)_2$ (prepared to solution by weight 0.173 mg of $Ca(OH)_2$ in 100 ml of H_2O) were selected to be alkali solutions.

Experiment

Simplex lattice design was used to design alkali solution for mix proportion of geopolymer as equation 1.

$$N = \frac{(q+m-1)!}{m!\,(q-1)!} \tag{1}$$

Where N is number of geopolymer mixture with various alkali activated solution. a {q, m} simplex-lattice design for q components consists of points defined by the following coordinate settings: the proportions assumed by each component take the m+1 equally spaced values from 0 to 1. In this work, q and m equal to 3 and 3 respectively. The conditions of lattice are augment of the design with center point and augment of the design with axial points as shown in Fig. 1.

Synthesis geopolymer

Fly ash was ground until it passed sieve#.200 (75 microns). The particle sizes of fly ash were less than 75 microns. 10 molar NaOH (NH), Na_2SiO_3 (NS) and $Ca(OH)_2$ solution (CH) were added in the mixture as follow Table 1. $Ca(OH)_2$ was prepared to be solution by using solubility limit of $Ca(OH)_2$ (0.173 g/100 ml H_2O). The alkali activated solution to fly ash ratio of 0.5 was kept in the experiment. Then, the geopolymer samples were put in cylinder mold with 3 cm diameter and 6 cm height. After 24 hours, geopolymer samples were taken out from the demold and wrapped by plastic until testing date.

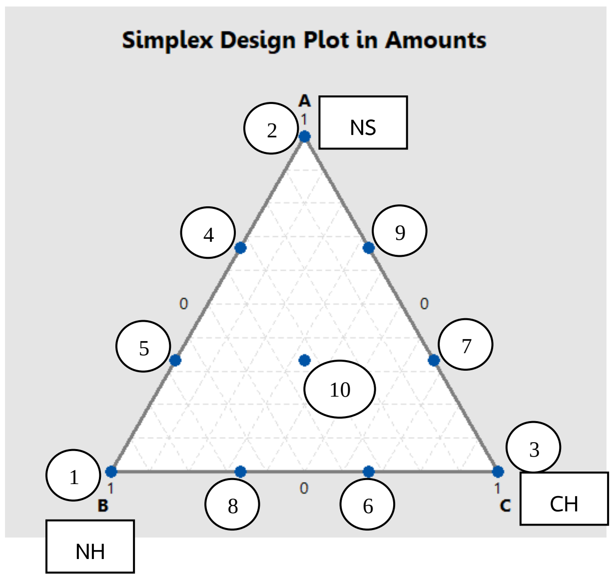

Fig. 1 Configuration of Design Runs for a {3,3} Simplex-Lattice Design

Table 1 Geopolymer mixture

No.	Fly ash (g)	NH solution (g)	NS solution (g)	CH solution (g)	symbol
1	1178	589	0	0	FANH
2	1178	0	589	0	FANS
3	1178	0	0	589	FACH
4	1178	395	196	0	FANH!NS
5	1178	196	395	0	FANHNS!
6	1178	395	0	196	FANH!CH
7	1178	0	395	196	FANS!CH
8	1178	196	0	395	FANHCH!
9	1178	0	196	395	FANSCH!
10	1178	196	196	196	FANHNSCH

Remark: calculated for cylinder mold with 3 cm diameter and 6 cm height for 20 samples

Testing

- Compressive strength of geopolymer was investigated at 7 14 and 28 days.
- The analysis of contour plot of the strength values was calculated by Minitab.
- The surface area was characterized by optical microscope technic.

Results and Discussions

Properties of starting materials

Table 2 shows chemical properties of fly ash. Fly ash has sum of SiO_2+Al_2O_3+Fe_2O_3 of 78.78% and high calcium of 13.82% indicating class F fly ash as described by ASTM C618 [12]. The rich in Si and Al are properly to be starting material for geopolymer.

The particle sizes distribution of fly ash are shown in Fig. 2. The median particle size of original fly ash was 67 microns. In order to improve reaction, fly ash was ground until it pass sieve#200. The median particle sizes were 12.44 microns. It was found that the small size of starting material has high surface area to react with alkali solution and provide the good mechanical properties of materials [2, 13].

Fig. 3 shows XRD pattern of fly ash. it composed of both amorphous phase and crystalline of Al_2O_3, SiO_2 and Al_2O_3 corresponding with chemical composition of fly ash.

Table 2 Chemical composition of fly ash

Composition (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
Fly Ash	39.47	29.46	9.85	13.82	-	3.66	-	1.95	1.8

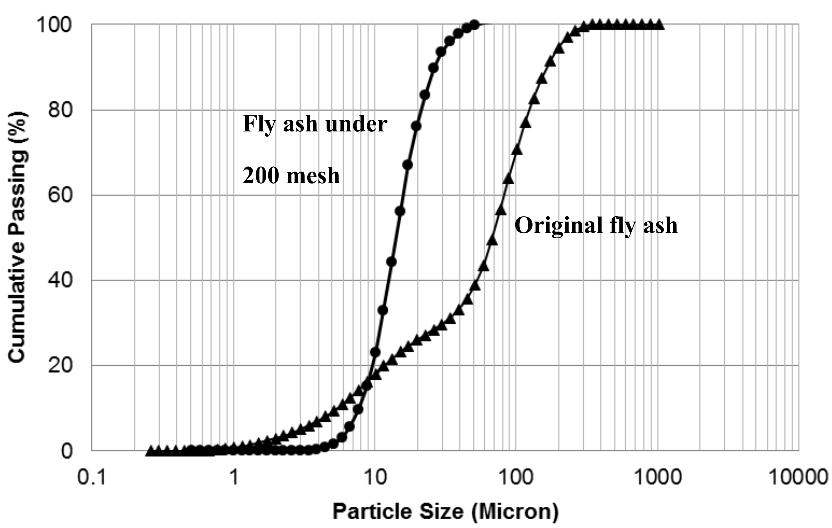

Fig. 2 Particle sizes distribution of original fly ash and fly ash under sieve #200

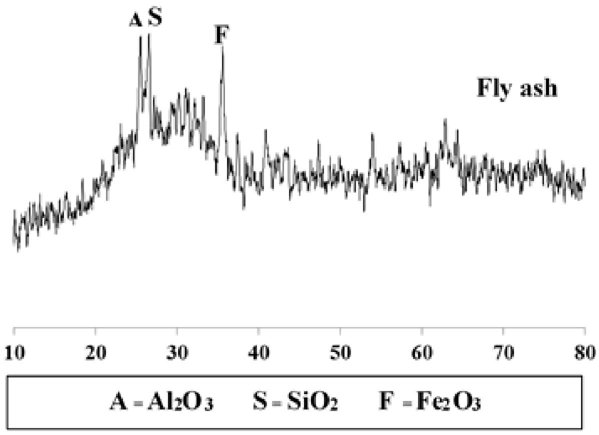

Fig. 3 XRD pattern of fly ash

Compressive strength of geopolymer

The compressive strength of geopolymer are shown in Fig 4. FANH!NS presented the highest compressive strength of 7.64 21.61 and 24.09 MPa at the age of 7 14 and 28 days, respectively. The highest compressive strength was due to high alkali hydroxide dissolute Si and Al from fly ash. In addition, Si from sodium silicate was rapidly react together with Si and Al from fly ash. The geopolymer reaction has high silica in the system to form geopolymer products [14]. FANHCH! showed the lowest compressive strength. High calcium hydroxide need time to react with Si and Al from fly ash and also low solubility in water. Nevertheless, calcium from $Ca(OH)_2$ can react with fly ash to form calcium silicate hydrate and enhance strength for geopolymer [9-11].

Predict strength of geopolymer and contour plot

Figure 5 shows The 4 in 1 residual plots for geopolymer with different alkali activated solution. The normal probability plot, versus fits, histogram, and versus order. It explained normal distribution of the data.

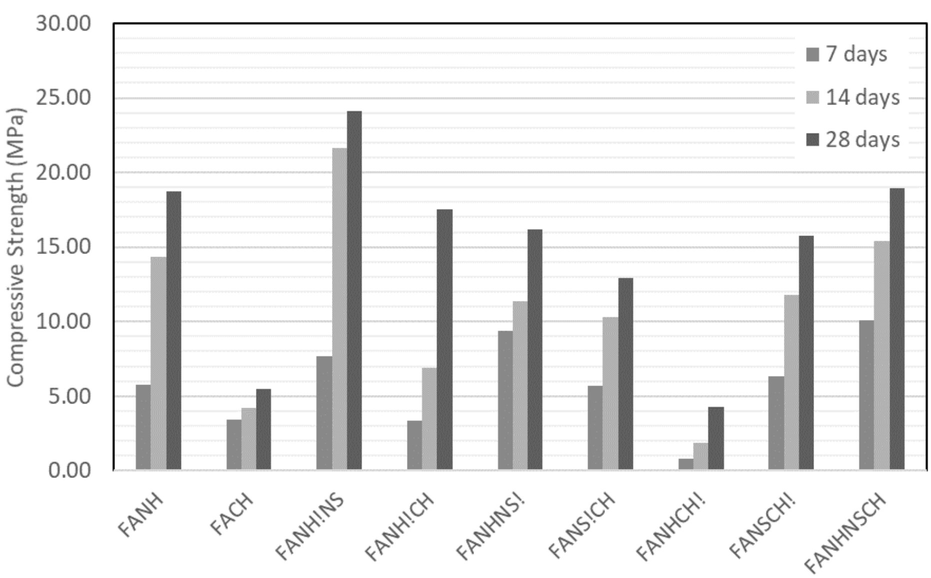

Fig. 4 Compressive strength of geopolymer with different alkali activated solution

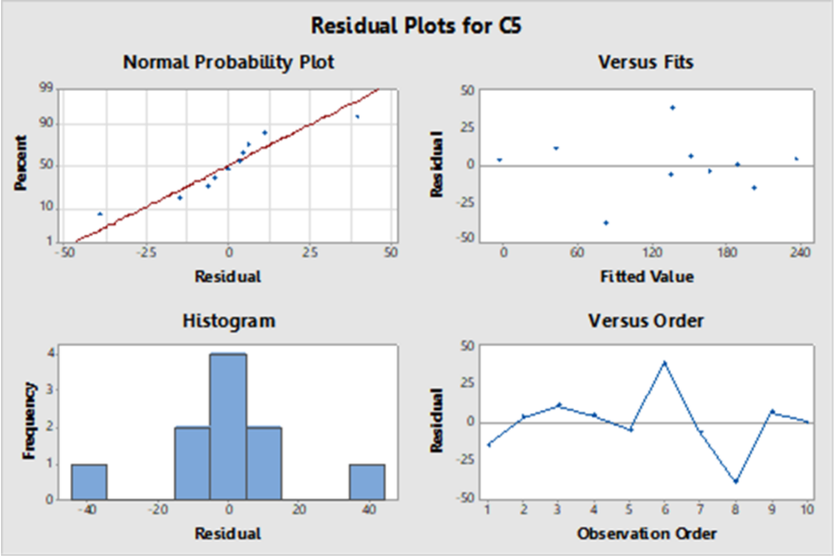

Fig. 5 4 in 1 residual plots for geopolymer with different alkali activated solution

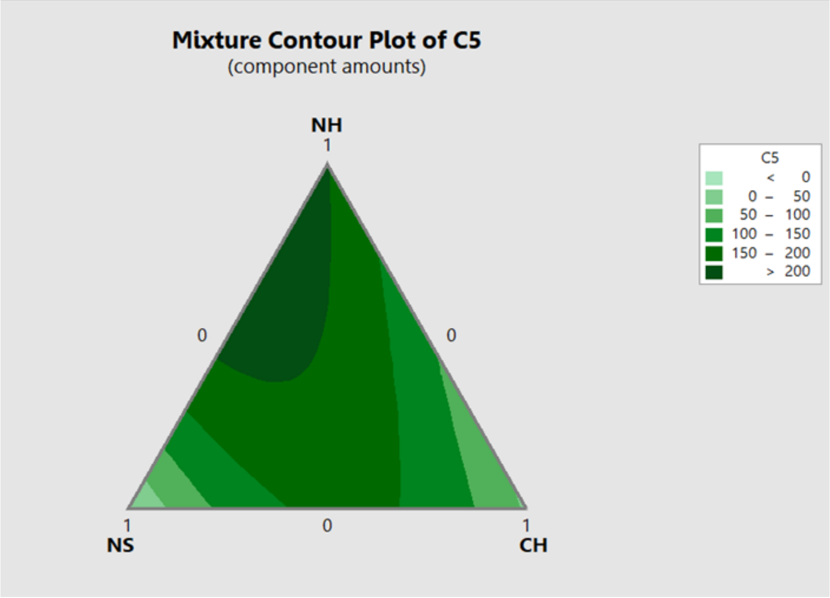

Fig. 6 Contour plot of geopolymer with different alkali activated solution

Figure 6 shows the contour plot of compressive strength of geopolymer in ksc unit with different three alkali activated solution, separated feasible area to 6 ranges for inhibition effect, less than 0, 0 to 50, 50 to 100, 100 to 150, 150 and 200 and more than 200 ksc. (20 MPa). This plot makes contrast as shown in Fig. 6. The darkest area is impossible area (more than 200 ksc) and lighter area is possible area for each range. It was found that geopolymer used NaOH and Na_2SiO_3 solution provided the highest compressive strength. Strong alkali hydroxide from NaOH can help dissolute Si and Al from fly ash. Moreover, SiO_2 from sodium silicate solution (Na_2SiO_3) is soluble Si which readily react with Si and Al from fly ash to form geopolymer products.

Microstructure of geopolymer

Fig. 7 presents surface analysis of geopolymer with different alkali activated solution by optical microscope (OM). The results showed that the geopolymer surfaces using different alkaline solutions were inhomogeneous. The surface of the geopolymer pastes were dense, no cavities, corresponding to the effect of compressive strength that the geopolymer using different alkaline solutions can obtain compressive strength. The surface of pastes were found the remaining unreactive fly ash. On the surface, a portion of the fly ash cavity that was removed from the reaction with the solution was also found.

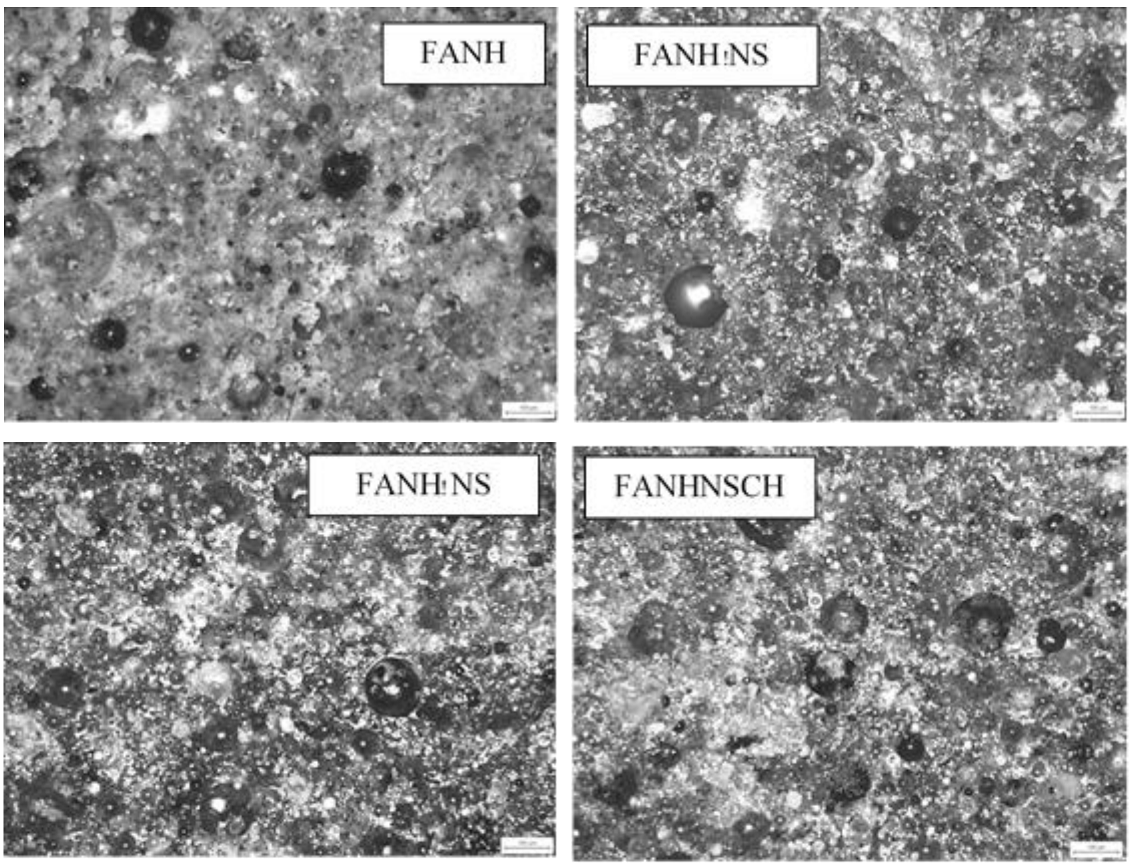

Fig. 7 Surface analysis of geopolymer with different alkali activated solution by optical microscope (OM)

Conclusion

Based on the study results, it can be concluded as follows:

1. Sodium hydroxide is the good alkali activated solution to leach Si and Al from fly ash.

2. Sodium silicate solution is the alkali solution which composed of readily dissolve Si and alkali solution in from Na_2O to dissolve Si and Al.

3. The geopolymer pasted with NaOH and Na_2SiO_3 present the highest compressive strength.

4. The equation for predicting the compressive strength in ksc of geopolymer with three alkali-activated solutions is Y = 202.49NH-3.63NS+43.20CH+461.24(NH*NS)-61.81(NH*CH)+559.15(NS*CH)+45.98(NH*NS*CH) where Y is compressive strength of geopolymer in ksc.

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